

**AMENDMENTS TO THE CLAIMS**

This listing of claims will replace all prior versions and listings of claims in the application:

**LISTING OF CLAIMS:**

**1-4. (canceled).**

**5. (currently amended):** The crystal according to claim 1, A crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1cyclohexylmethyl)-9-(4-(4-carboxyphenyloxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride which has a powdery X ray diffraction spectrum shown in Fig. 1.

**6. (original):** The crystal according to claim 5, which has diffraction angle  $2\theta$  of 5.15, 8.06, 10.26, 11.01, 13.72, 15.46, 17.36, 18.03, 18.58, 19.00, 19.51, 20.71, 21.73, 22.58, 23.80, 24.96 and 27.07(degree) on the powdery X ray diffraction spectrum.

**7. (currently amended):** A crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1cyclohexylmethyl)-9-(4-(4-carboxyphenyloxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride~~The crystal according to claim 1,~~ which has an IR absorption spectrum shown in Fig. 3.

**8. (original):** The crystal according to claim 7, which has absorptions at 2924, 2504, 1682, 1632, 1597, 1503, 1426, 1377, 1235, 1163, 1098, 961, 928, 876, 855, 770, 727 and  $681\text{ cm}^{-1}$  on the IR absorption spectrum.

**9. (currently amended):** The crystal according to claim 45 or 7, which has a mean particle size of about 0.05  $\mu\text{m}$  to about 200  $\mu\text{m}$ .

**10. (currently amended):** The crystal according to claim 25 or 7, which is a crystal of  $P2_1$  space group.

**11. (original):** The crystal according to claim 10, which has lattice constants of  $a = 11.8105 \text{ \AA} \pm 7\%$ ,  $b = 7.8730 \text{ \AA} \pm 7\%$  and  $c = 18.2351 \text{ \AA} \pm 7\%$ .

**12. (canceled).**

**13. (original):** A process for producing a crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride, which comprises carrying out crystallization from a lower alcohol solvent which may contain water or a water-miscible ether solvent which may contain water, in which a crudely purified substance of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride is dissolved or suspended.

**14. (original):** The process according to claim 13, wherein the lower alcohol solvent is  $C_{1-4}$  alkyl alcohol or  $C_{1-4}$  alkyl acetate.

**15. (original):** The process according to claim 14, wherein the lower alcohol solvent is methanol or ethanol.

**16. (original):** The process according to claim 14, wherein the lower alcohol solvent is ethyl acetate.

**17. (original):** The process according to claim 13, wherein the water-miscible ether solvent is 1,2-dimethoxyethane, dioxane or tetrahydrofuran.

**18. (original):** The process according to claim 13, wherein the water and the lower alcohol solvent or the water and the water-miscible ether solvent are mixed in a mixing volume ratio of 1 : 50 to 7 : 3.

**19. (original):** The process according to claim 18, wherein the water and the lower alcohol solvent or the water and the water-miscible ether solvent are mixed in a mixing volume ratio of 1 : 35 to 5 : 5.

**20. (original):** The process according to claim 13, wherein the crystallization is carried out at about -10°C to about 40°C.

**21. (original):** The process according to claim 13, wherein the crystallization is carried out for about 20 minutes to about 5 hours.

**22. (currently amended):** A crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxy-phenyloxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride which is obtainable by the process according to claim 13

which has a powdery X ray diffraction spectrum shown in Fig. 1 and which has an IR absorption spectrum shown in Fig. 3.

**23. (original):** A process for producing a crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride, which comprises: dissolving or suspending (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane and hydrogen chloride in a solvent selected from (1) C<sub>1-4</sub> alkyl alcohol, (2) a mixed solvent of C<sub>1-4</sub> alcohol and water, (3) a water-miscible ether solvent, (4) a mixed solvent of a water-miscible ether solvent and water, (5) a mixed solvent of C<sub>1-4</sub> alkyl alcohol and a water-miscible ether solvent, (6) a mixed solvent of C<sub>1-4</sub> alkyl alcohol, a water-miscible ether solvent and water and (7) water, followed by heating at about 40°C to about 80°C; and cooling the resulting mixture at about -5°C to about 35°C.

**24. (currently amended):** The process according to claim 23, wherein the C<sub>1-4</sub> alkyl alcohol is methanol or ethanol.

**25. (original):** The process according to claim 23, wherein the water-miscible ether solvent is 1,2-dimethoxyethane, dioxane or tetrahydrofuran.

**26. (currently amended):** The process according to claim 23, which comprises: dissolving or suspending (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane and hydrogen chloride in a solvent selected from (1) C<sub>1-4</sub> alkyl alcohol, (2) a mixed solvent of C<sub>1-4</sub> alkyl alcohol and water,

(3) a water-miscible ether solvent, (4) a mixed solvent of a water-miscible ether solvent and water, (5) a mixed solvent of C<sub>1-4</sub> alkyl alcohol and a water-miscible ether solvent, (6) a mixed solvent of C<sub>1-4</sub> alkyl alcohol, a water-miscible ether solvent and water and (7) water, followed by heating at about 40°C to about 80°C; cooling the resulting mixture at about -5°C to about 35°C; adding C<sub>1-4</sub> alkyl alcohol or a water-miscible ether solvent to the mixture; and optionally adding water to the mixture.

**27. (original):** A process for producing a crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride, which comprises dissolving or suspending a solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride or amorphous (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride in C<sub>1-4</sub> alkyl acetate, followed by heating at about 40°C to about 80°C; and cooling the resulting mixture at about -5°C to about 35°C.

**28. (currently amended):** A crystal of a non-solvate of (3R)-1-butyl-2,5-dioxo-3-((1R)-1-hydroxy-1-cyclohexylmethyl)-9-(4-(4-carboxyphenoxy)phenylmethyl)-1,4,9-triazaspiro[5.5]undecane hydrochloride which is obtainable by the process according to claim 23 which has a powdery X ray diffraction spectrum shown in Fig. 1 and which has an IR absorption spectrum shown in Fig. 3.

**29. (original):** The crystal according to claim 28, which has a mean particle size of about 0.05  $\mu\text{m}$  to about 200  $\mu\text{m}$ .

**30-34. (canceled).**

**35. (new):** The crystal according to claim 5 or 7, which has a melting point of about 230°C to about 240°C.

**36. (new):** The crystal according to claim 5 or 7, which has a melting point of about 232°C to about 235°C.